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NSWC TR 90-48

A SILVER-BEARING, HIGH-TEMPERATURE, SUPERCONDUCTING (HTS) PAINT

BY WILLIAM A. FERRANDO

RESEARCH AND TECHNOLOGY DEPARTMENT

15 FEBRUARY 1990

NAVAL SURFACE WARFARE CENTER

Dahlgren, Virginia 22448-5000 • Silver Spring, Maryland 20903-5000

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FOREWORD

While the desirability of producing the high-temperature superconducting (HTS) materials in wire form is undeniable, there is a subgroup of devices which might be improved significantly if the superconductor were available as a thick film or paint with appropriate properties. Such a paint could be applied by brush or spray and heat treated to produce the superconducting properties. Paint coatings under discussion here fall within the range of about 10-100 microns. Potential applications of thick film coatings include infrared detectors, magnetic field sensors, magnetic shielding, and microwave cavities.

The HTS paint system described here is capable of providing a fully superconductive coating at 77K with the additional benefits of good bonding to the substrate, resistance to microcracking, and environmental protection.

The author wishes to acknowledge Mr. Dave Divecha of the Naval Surface Warfare Center (NSWC) for his encouragement in this investigation. The work was supported by NSWC Independent Exploratory Development (IED) funds.

Approved by:

Carl E. Mueller

CARL E. MUELLER, Head
Materials Division

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ILLUSTRATIONS

CHAPTER 1

INTRODUCTION

For the initial promise of the high-temperature superconducting (HTS) materials to be sustained, it is imperative that some early application be made. It is likely that this will take the form of component modification of an already existing system or device to accommodate the superconductor. It seems obvious, also, that early applications should not rely on extreme material properties such as very high critical temperature (T_c) or current (J_c). Likewise, an early application involving a long, ductile wire of these materials may be unrealistic.

There is, however, a class of potential applications for HTS materials in the form of a paint coating on suitable substrates and in appropriate geometries which may not rely on the extremes of HTS material properties. Those of potential near-term interest to the Navy are described below. The paint method proposed is a response to these reported needs.

CHAPTER 2

BACKGROUND AND APPLICATIONS

INFRARED DETECTORS

This class of detector depends upon the photoconductive properties of the film. Current infrared (IR) detector technology includes Hg-Cd-Te cooled to 77K and doped Ge cooled to 4.2K. These materials are expensive and difficult to configure in arrays. The photoconductive carrier lifetimes in these materials must be reduced by doping to obtain the proper frequency response. This doping, however, tends to reduce sensitivity and to increase noise and stray capacitance of the film.

HTS films have a natural network of grain boundaries which can cause problems in some applications, but which may be exploited to advantage in this case. Films of HTS materials having prominent grain boundary configuration show a large reduction of voltage (resistance) upon illumination with light.[1] Thus, since the required type of photo response has been obtained with HTS materials for this application, materials optimization and fast detector development remain.

GRADIOMETER AND MAGNETOMETER SENSORS

Superconducting Quantum Interference Device (SQUID) magnetometers are the most sensitive measuring devices of magnetic fields known. SQUIDs operating at 4.2K have a theoretical sensitivity of about $2X10^{-7}$ gauss. HTS SQUIDs have been developed with laboratory demonstrated performance at 77K equal to that of commercial SQUIDs at 4.2K utilizing conventional niobium (Nb) technology.[2] The highest sensitivity, after material and design optimization, always will occur at liquid helium temperature. This is due to the reduction of Johnson noise and thermally induced magnetic noise in the normal metals present and the more desirable magnetic properties of helium itself. Microscopically uniform addition of silver may impart a higher density, degree of coupling, and uniformity to the HTS particulate coatings. This added to an oriented grain structure may provide the coating medium required for improved performance in such devices.

MAGNETIC SHIELDING AND WAVEGUIDE CAVITY COATINGS

Although they are substantially different applications, both magnetic shielding and waveguide cavities depend upon the "skin" or "penetration depth" to relate to the other coating properties. This is a measure of the depth a magnetic field of given magnitude and frequency will penetrate a superconducting surface. This depth is also a function of the "reduced" temperature of the superconductor, T/T_c. For shielding applications, the film thickness should be greater than the penetration

depth. For waveguide applications, the penetration depth is a measure of losses as the signal is transmitted along the tube. The depth must be minimized for highest efficiency or "Q". The presence of finely deposited and highly bonding silver should help in bridging microcracking, relieving stresses, and minimizing chemical reaction with the substrate which have been encountered in previous efforts.[3] Finally, silver addition was found to significantly reduce environmental degradation of HTS films due to incursion and reaction of moisture along grain boundaries.[4]

CHAPTER 3

FORMULATION, APPLICATION, AND PROCESSING OF THE COATING

The chemical reactions and phase changes of $AgNO_3$ which allow it to be used in the context of micro-coating of ceramic superconductor particles were discussed previously.[5] The thermal behavior of $AgNO_3$ is:

$$AgNO_3 \text{ -----> } AgNO_3 \text{ (liquid)} \qquad @\,222°C$$

$$2AgNO_3 \text{ -----> } 2AgO + 2NO_2\uparrow \text{ (brown fumes)} \qquad @\,444°C$$

$$2AgO \text{ -----> } 2Ag + O_2$$

or perhaps more correctly:

$$2AgNO_3 \text{ -----> } 2Ag + O_2 + 2NO_2 \qquad @\,444°C$$

Results on bulk superconductors indicate the decomposition of $AgNO_3$ to provide a superior method of Ag addition.

The formulation of an HTS paint using $AgNO_3$ is rather simple. Figure 1 outlines the process. $AgNO_3$ is ground and added to the HTS powder. ($YBa_2Cu_3O_{7-x}$, designated 123, was used in this work, but another HTS compound could be substituted.) Uniform fine particle (small compared with the coating thickness) HTS powder should be used. Generally, a quantity of $AgNO_3$ is added to produce about 15wt % after decomposition. Sufficient pure ethylene glycol is added to provide a good consistency for brushing (or spraying). The glycol is inert to the 123 compound and dissolves the $AgNO_3$, spreading it uniformly throughout the entire mass. Solubility of $AgNO_3$ in glycol was found to be at least 7.5 g/10 cc, a fraction greater than would ever be needed in an HTS paint.

The suspension mixture is applied to the substrate by brushing or spraying. The choice of substrate is critical for successful HTS coating. The substrate must be able to tolerate the sinter temperature without cracking or chemically reacting with the HTS compound. Its thermal expansion must match that of the superconductor, so that the film remains under compression during thermal cycling. This retards the formation of microcracks which impair J_c and broaden the superconducting transition. The substrate must not produce too great a compression on cooling, however, or separation of the film could occur.

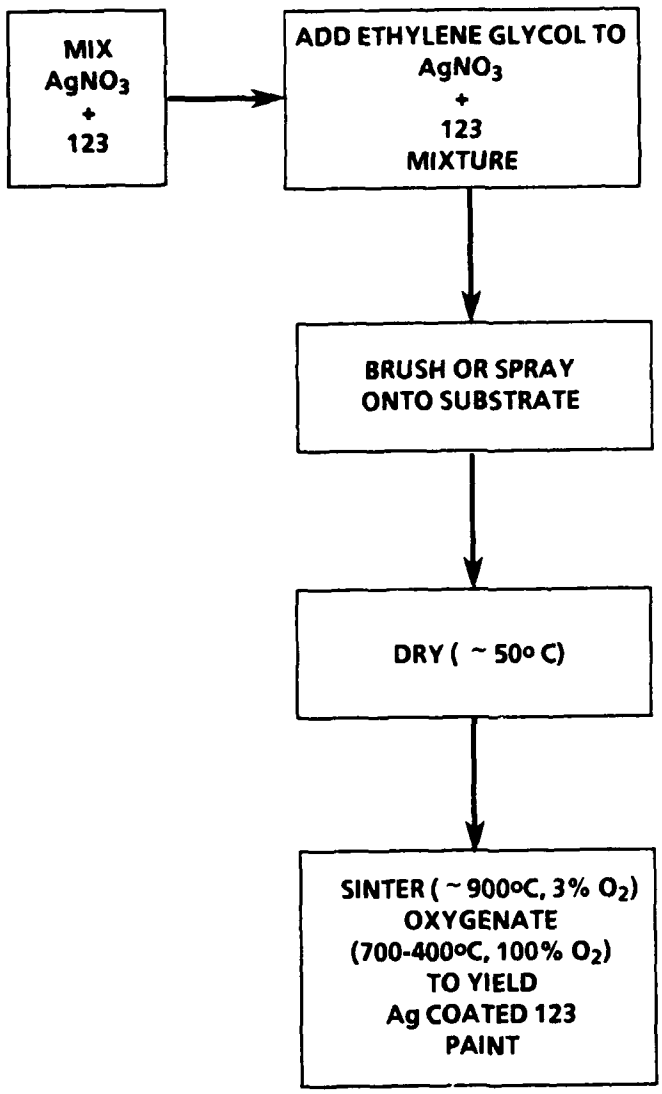

FIGURE 1. FLOW CHART FOR PRODUCTION AND APPLICATION OF GLYCOL-BASED 123/$AgN0_3$ PAINT

SUBSTRATE REQUIREMENTS

Various substrates have been tested in the course of several HTS thin film efforts.[3,4] These include Ba-Ti, Ni-Al-Ti, Al_2O_3, spinel, MgO, SiO_2, $SrTiO_3$, and YSZ (yttria stabilized zirconia). Those containing nickel (Ni) or titanium (Ti) experienced some degree of chemical reaction. The most successful substrates were those of spinel and YSZ. These yielded complete superconducting transitions with T_c of about 81K and transition widths of less than 10K.

CHAPTER 4

EXPERIMENTAL PROCEDURE AND RESULTS

A 2-inch diameter tube of Al_2O_3 was brush painted with the 123/$AgNO_3$/glycol mixture and dried at about 50°C (Figure 1). The tube was subsequently fired at ~460°C to decompose the $AgNO_3$. Care was taken to completely evaporate the glycol prior to firing; otherwise, the resulting combustion will alter the final coating properties. This operation was carried out several times to produce a coating thickness of several mils. The tube was sintered overnight at 900°C in O_2. Four contacts were attached using silver paint. Measurement of T_c was made using the apparatus and technique described in Reference 5.

A sharp drop in resistance was observed at about 85K; however, a small residual resistance remained at 77K. The curve remained substantially unchanged with further annealing of the tube in O_2. This indicated the possibility of unhealed microcracks in the coating due to mismatch in thermal expansion with the tube. The curved tube presented difficulties for microscopic observation. Thus an alumina flat was identically coated and heat treated for microscopic study. Figure 2 shows the painted surface (top) at high magnification and the Ag elemental map (bottom). This shows the degree of Ag dispersion achievable by the $AgNO_3$ decomposition method. The distribution is remarkable for its homogeneity and microparticulate character.

Available thoria (ThO_2) tube sections were tested as an alternative substrate material. To help obviate the problem of electrical contact, four silver metal strips were bonded along the inside of the thoria tube using small amounts of $AgNO_3$ prior to painting. Then the tube inside was painted with the 123-$AgNO_3$-ethylene glycol mixture. The ethylene glycol was allowed to evaporate at ~50°C. The tube was transferred to a furnace, sintered in O_2 for 16 hours at 900°C, and allowed to cool in the furnace to ambient.

Electrical connections (visible in the picture) were made to the ends of the silver tabs now embedded in the coating. T_c measurement was made by slowly lowering the tube into a liquid nitrogen dewar while monitoring the coating resistance with a Keithley Model 503 milliohmmeter. Temperature and resistance were recorded using a Houston Instruments dual pen recorder. Figure 3 shows the tube section with attached four contact leads and mounted on a four-pin plug assembly. The data is shown plotted in Figure 4. A complete superconducting transition of several degrees width and T_c of about 81K was observed (circle points). After thermal cycling and several months exposure to ambient conditions, during which no particular precautions to protect the tube were taken, a second measurement was made. This set of data (square points) indicates essentially the same superconducting behavior. Hence, a reasonably stable paint film, fully superconducting at 77K, has been applied. An optimally stable HTS paint coating will depend strongly on the phase purity of the 123 starting material and the high degree of sealing of the HTS grain boundaries against moisture by the Ag (see Reference 4).

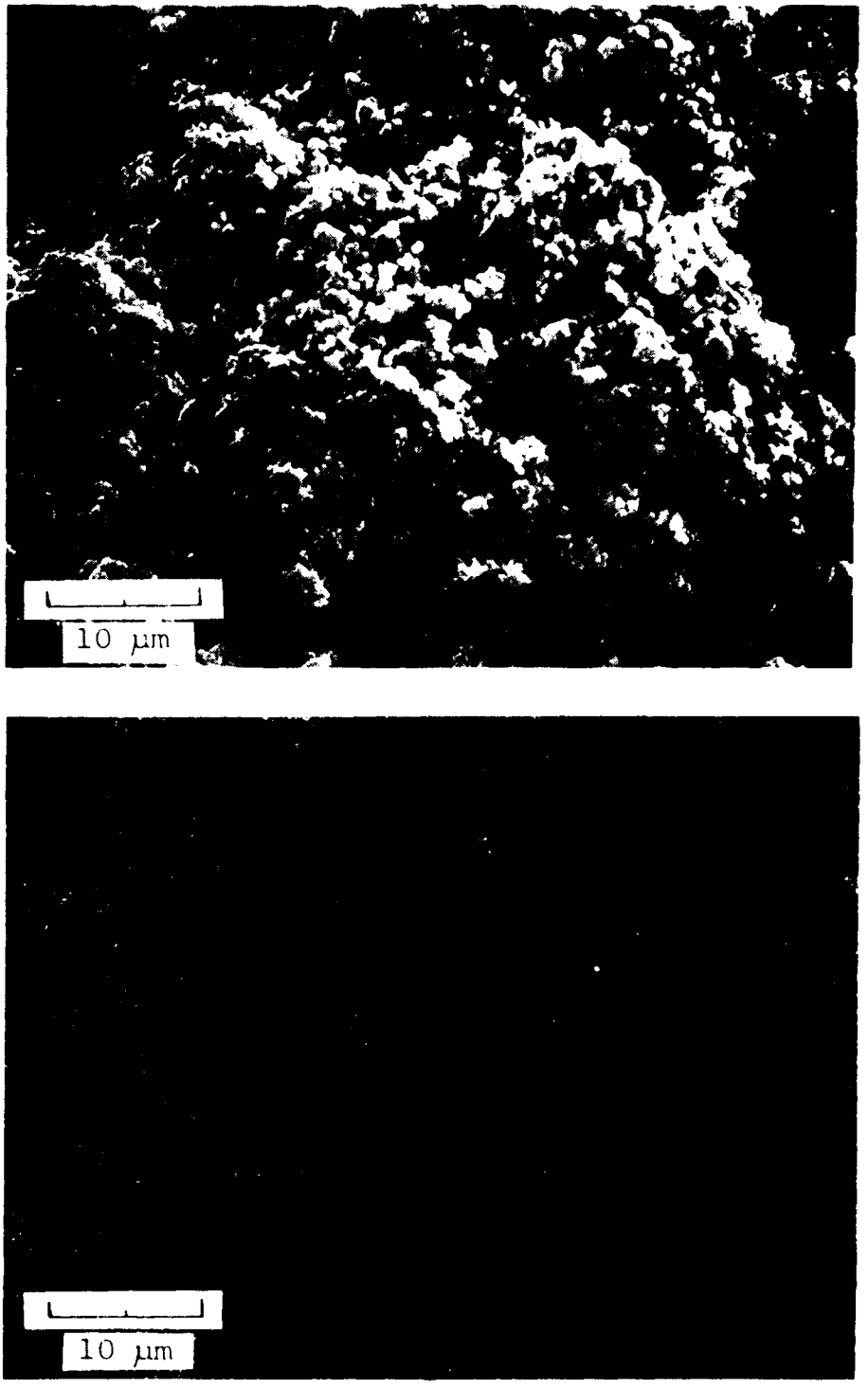

FIGURE 2. SEM PHOTOGRAPH OF 123/Ag PAINTED SURFACE (ALUMINA FLAT) WITH Ag DISTRIBUTION MAP

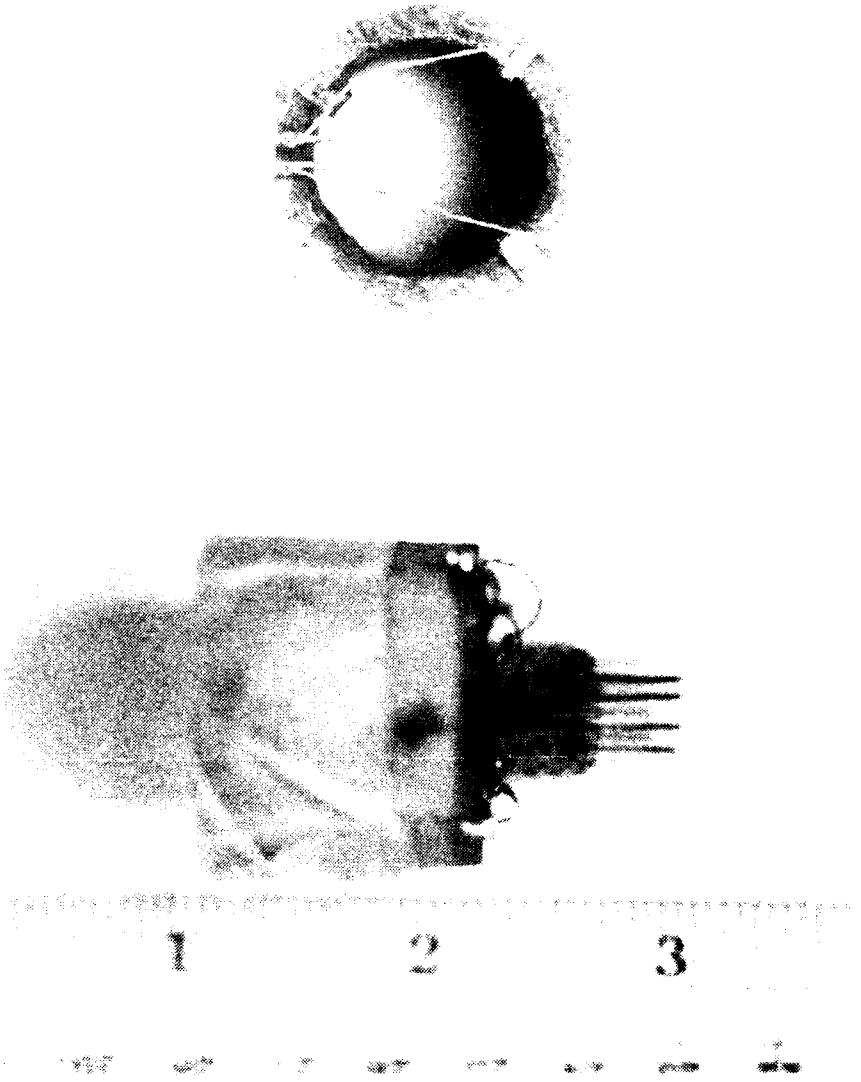

FIGURE 3. PHOTOGRAPH OF PAINTED THORIA TUBE TEST SAMPLE MOUNTED ON LOW-TEMPERATURE PROBE PLUG

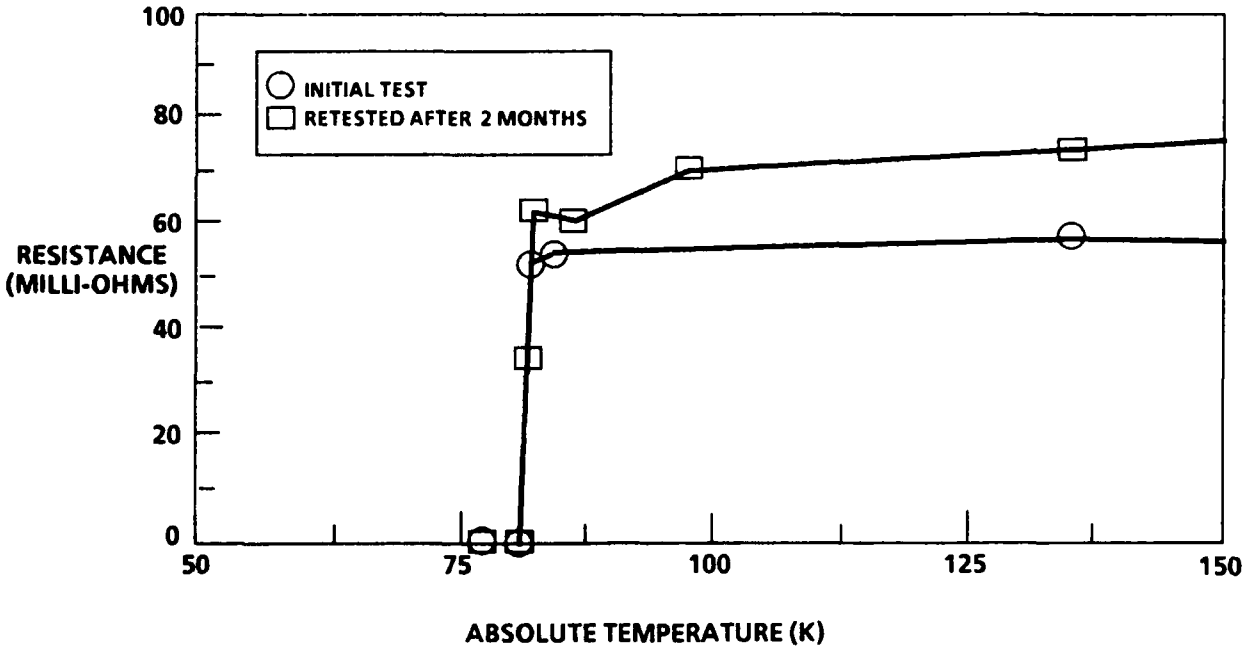

FIGURE 4. ABSOLUTE TEMPERATURE VERSUS RESISTANCE OF THORIA TUBE COATED WITH 123/$AgN0_3$ PAINT AFTER THERMAL PROCESSING

A crude measurement of J_c for this film was made. The estimated paint thickness is approximately equal to that of the embedded Ag contact strips which were ~.005 inch (.013 cm). The tube length was ~1 inch (2.5 cm). The tube carried about 0.01 amps in the superconducting state. Thus, the J_c of the paint cross section may be computed as $J_c = .01/(.013 \times 2.54) = .30$ A/cm^2. While some applications of HTSC coatings might not need a substantial J_c, most involving microwave handling will require at least moderately high J_c.

Proper choice of substrate and sintering/oxygenation heat treatment schedule[6] should produce the improvement required for the HTS paint applications noted above. Other HTS compounds also can be used with the $AgNO_3$ decomposition process to produce paint films. The presence of the thoroughly dispersed and intimately bonded Ag should reduce the incidence of microcracking, leading to good thermal cycling performance.

CHAPTER 5

SUMMARY

A well-bonded, environmentally stable superconducting paint film containing Ag has been produced on the inside surface of a thoria tube. A method of $AgNO_3$ decomposition was used for Ag dispersion in the 123 superconductor material and for bonding with an ethylene-glycol vehicle for application. This film displays full superconductivity at 77K and retains its properties for an extended time under ambient unprotected conditions. Improvement upon these results certainly is likely using other substrates and more careful heat treatment.

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13. ABSTRACT *(Maximum 200 words)*

A substantial set of device applications awaits development of a workable, durable, high-temperature superconducting (HTS) paint. Such a paint should be truly superconducting with its critical temperature $T_c > 77K$. For most of these applications, a high critical current (J_c) is not required, although probably desirable.

A process is described which can be used to produce silver-bearing HTS paint coatings on many engineering materials. Preliminary tests have shown good adherence to several ceramics and the ability to meet the superconducting criteria. Moreover, the coatings withstand multiple thermal cycling and stability under laboratory ambient storage conditions for periods of at least several months.

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Blocks 17.-19. Security Classifications. Self-explanatory. Enter U.S. Security Classification in accordance with U.S. Security Regulations (i.e., UNCLASSIFIED). If form contains classified information, stamp classification on the top and bottom of the page.

Block 20. Limitation of Abstract. This block must be completed to assign a limitation to the abstract. Enter either UL (unlimited) or SAR (same as report). An entry in this block is necessary if the abstract is to be limited. If blank, the abstract is assumed to be unlimited.